

# IMPROVED QUANTITATION OF SEMIVOLATILE COMPOUNDS BY EXTRACTED STANDARDS

RICK McMILLIN, DIANE GREGG, LISA HURST,  
U.S. Environmental Protection Agency, Region 6 Lab,  
10625 Fallstone Rd., Houston, TX. 77099

***Note:** This paper was presented at Pittcon'96, Chicago, IL, March 5, 1996, as paper #492. Material presented reflects the opinions of the authors only and do not reflect EPA opinion, policy, or endorsement.*

## **ABSTRACT:**

It is important to report data for environmental samples as accurately as possible to properly assess public health dangers. Current EPA Semivolatile methodologies typically give less than 100% recoveries because they do not correct for inherent extraction inefficiencies. This can result in low bias results being reported routinely due to uncorrected recoveries. Some Semivolatile methods allow recovery of certain compounds to be as low as 10% and still be acceptable<sup>1,2</sup>. This could result in a value being reported in a sample that is 1/10<sup>th</sup> of the true value, which could be a significant difference. The one EPA method for Semivolatiles that corrects for extraction recovery is method 1625<sup>3</sup>, isotopic dilution, but this method has been considered too complex or expensive to implement by most laboratories and is not commonly used. This paper investigates simple and inexpensive alternatives.

For some variations in this study, extraction recoveries have been compensated by extracting a calibration standard for quantitation purposes -- with the objective being to improve real sample quantitation accuracy. In addition, internal standards have been added to some samples before they were extracted to partially compensate for individual matrix or extraction effects. Theoretically, quantitation using these modifications should give 100% recovery for all analytes independent of extraction technique (assuming good precision within a set). This should improve correlation between reported amount and true sample concentration.

DI water, ground water, waste water, and soil matrices were evaluated and compared using the following variations for quantitation:

- **(variation 1)** Current EPA method unchanged (quant standard NOT extracted, ISTDs added AFTER sample extraction).

---

<sup>1</sup>EPA CLP SOW, OLM03.0

<sup>2</sup>EPA SW-846, "Test Methods for Evaluating Solid Waste", update II

<sup>3</sup>40CFR, 136, Appendix A

- ▶ **(variation 2)** Current EPA method, but using extracted ISTDs (quant standard NOT extracted)
- ▶ **(variation 3)** Current EPA method, but using extracted quantitation standard (ISTDs added AFTER sample extraction)
- ▶ **(variation 4)** Current EPA method, but using extracted ISTDs and extracted quantitation standards.

Water samples were extracted using Accelerated One-Step™ and soil samples were extracted by automated soxhlet. Analysis was performed by GC/MS method 8270. Results are listed below

PERCENT RECOVERY*				
	DI water 10ug/L	Grd Water 10ug/L	W Water 10ug/L	Soil 1000ug/Kg
<b>Variation 1</b>	65%	69%	74%	56%
<b>Variation 2</b>	84%	91%	89%	81%
<b>Variation 3</b>	84%	86%	94%	71%
<b>Variation 4</b>	96%	98%	96%	93%

\*Mean for 73 analytes, including 8 surrogates, 6-7 replicates

## Conclusions

The major disadvantage to Variations 2 and 4 is with samples that may require dilutions. The extracted internal standard would make it impossible to dilute the sample relative to the internal standard. More internal standard could not be added to the extract and maintain the advantage of adjusting for extraction effects. This would require re-extractions at reduced volume, which may not have been required with the original method. Also, an internal standard might be affected more by the extraction process than the associated analytes, thus reducing the accuracy of the quantitation for all of those analytes. Variations 2-4 will also have significantly more high bias than Variation 1, but the mean should be closer to true value.

In spite of those drawbacks, this data shows that there is room for improvement in the accuracy of Semivolatiles by current methods. The use of method variation 4 has already been proven by the many Volatiles methods currently in use (although re-extractions are normally just a simple purge and trap re-analysis). Variation 2 has been proven by the EPA drinking water method 525.2<sup>4</sup> and others. Variation 3 has been proven by the EPA drinking water methods 504.1<sup>4</sup> and 515.2<sup>4</sup>. These methods use an extracted standard called a “process standard” for

---

<sup>4</sup>“Methods for the Determination of Organic Compounds in Drinking Water”, EPA/600/4-88/039, including supplements II and III.

quantitation. Method 525.2 already requires an extracted standard, though it isn't used for quantitation (laboratory fortified blank). Other methods also have an extracted standard which is called a lab QC sample. Therefore it would be no extra expense or effort to use these as quantitation standards. That would compensate for the most basic inherent extraction inefficiencies of the method and improve accuracy. Is it more important to know the exact amount of analyte in the extract, or the concentration of the analyte in the sample? Less reliance on extraction efficiency, by using Variations 2-4, could lead to more innovation in the extraction process and produce more accurate results for Semivolatile analysis.